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IS 13160 (1991): Aleuritic Acid [CHD 23: Lac, Lac Products and Polishes]

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Lac and Lac Products Sectional Committee, CHD 022

## FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Lac and Lac Products Sectional Committee had been approved by the Chemical Division Council.

Aleuritic acid is derived from lac and is principally used in the perfumery industry. It is an important raw material for synthesis of macrocyclic musk like lactons such as ambrettolide, civetone, etc.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values ( *revised* ).' The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# AMENDMENT NO. 1 FEBRUARY 2007
# TO
# IS 13160 : 1991 ALEURITIC ACID — SPECIFICATION

(*Page* 1, *clause* **4**) — Substitute the following for the existing:

**'4 REQUIREMENTS**

**4.1 Mandatory Requirements**

The material shall be off white in colour and shall comply with the requirements as given in Table 1.

**4.2 Optional Requirements**

Purity level of the material shall be as mutually agreed to by the purchaser and the supplier as ascertained by DSC analysis as outlined in **A-7**.'

(*Page* 2, *Annex* A) — Insert the following at the end:

## A-7 TESTING OF PURITY USING DIFFERENTIAL SCANNING CALORIMETER (DSC)

### A-7.1 General

The purity of aleuritic acid (9, 10, 16 trihydroxyhexadecanoic acid) can be determined from its melting profile (a plot of heat flow vs. temperature) using a Differential Scanning Calorimeter. The information about the above is obtained by analyzing the melting profile in a computer through appropriate software. A DSC also provides information about the melting temperature, a graphical presentation of the melting peak of the acid and presence of impurity, if any. The melting profile provides instantaneous information about the relative homogeneity/crystallinity of the material.

### A-7.2 Apparatus

a) A Differential Scanning Calorimeter with different rates of heating, computer controlled, with software for peak position, selection of ranges of dependent and independent variables, onset temperature, purity, percentage solid, partial area, etc.

   The temperature range required for the analysis of aleuritic acid is 40-150°C.

b) Printer, printing papers for the output from DSC analysis*

c) Aluminium pans with cover for DSC analysis with crimper*

d) A good weighing balance with accuracy of ± 0.01 mg.

*The equipment/material would normally be provided with DSC.

### A-7.3 Procedure

**A-7.3.1** Weigh finely powdered 5-6 mg of aleuritic acid, accurately, in the aluminium pan, put the cover and crimp properly.

**A-7.3.2** Switch on the printer, DSC (precalibrated) and controller. Start purging the sample holder with Ar or $N_2$. Start the compressor/circulator.

**A-7.3.3** Place the sample in the crimped pan in the sample holder of DSC.

**A-7.3.4** Start the software, select the appropriate menu for a temperature profile run. Enter the sample, file name, etc. Enter start and end temperatures, run rate (20°C/min), etc.

**A-7.3.5** After attaining the steady state, start run, save the profile after completion of the run.

**A-7.3.6** Enter into analysis software, select the appropriate portion of the profile, preferably containing the initial and final plateau regions.

### A-7.4 Examination of the Melting Profile

Pure crystalline aleuritic acid gives sharp single melting peak. Presence of impurity gives broad peak associated with smaller peaks, shoulders, etc. Broader peak is indicative of heterogeneity.

The onset temperatures for threo and erythroisomers of aleuritic acid were observed to be 94.9 and 119.3°C, respectively. The data are based on samples crystallized in water.

### A-7.5 Analysis of the Profile

**A-7.5.1** *Melting Temperature*

Position of the melting peak will be shown on the screen, which can be taken as melting temperature. In case of good crystalline material, melting peak will be obtained as mentioned in **A-7.4.** Melting temperature will depend on method of crystallization. In case broad melting peak or more than one peak is obtained, further crystallization of the sample is to be done till sharp single peak is obtained.

**A-7.5.2** *Purity*

Open the Purity menu. Enter the weight of the sample taken and its molecular weight. Select proper regions. Enter the purity calculation. Repeat the operation for different regions. Select purity values from a set of near similar values.

### A-7.6 Report

The report shall contain position of the peak (melting temperature) in degrees Celsius, purity (%), for multiple peaks, the number of peaks with their positions. The sample shall be considered passing if the nature of peak and purity level is as per those agreed to, mutually, by the purchaser and the supplier.

( CHD 23 )

Reprography Unit, BIS, New Delhi, India

# *Indian Standard*

# ALEURITIC ACID - SPECIFICATION

## 1 SCOPE

This standard specifies requirements and methods of sampling and test for aleuritic acid.

## 2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

| *IS No.* | *Title* |
|---|---|
| 1070 :1977 | Specification for water for general laboratory use ( *second revision* ) |
| 2362 : 1973 | Determination of water by the Karl Fischer method *( first revision )* |
| 4905 : 1968 | Methods for random sampling |
| 4908 : 1968 | Glossary of terms used in lac industry |
| 5762 : 1970 | Methods for determination of melting point and melting range |
| 6921 : 1973 | Methods of sampling and test for lac and lac products |

## 3 TERMINOLOGY

**3.1** For the purpose of this standard, the definitions given in IS 4908 : 1968 and the following shall apply.

**3.1.1** *Approved Sample*

The sample agreed upon between the purchaser and the supplier as the standard for its requirements.

## 4 REQUIREMENTS

The material shall be of white in colour and shall comply with the requirements as given in Table 1.

**Table 1 Requirements for Aleuritic Acid**

( *Clause* 4.1 )

| Sl No. | Characteristic | Requirements | Methods of Test, Ref to Cl No. In Annex A |
|---|---|---|---|
| (1) | (2) | (3) | (4) |
| i) | Moisture, percent by mass, *Max* | 1.0 | A-2 |
| ii) | Molecular mass ( from acid value ) | 303-308 | A-3 |
| iii) | Melting point, °C, *Min* | 96 | A-4 |
| iv) | Relative density, 27±2°C and 65±5 RH | 1.1 | A-5 |
| v) | Infra-red spectrum | Close match to the approved sample | A-6 |

## 5 PACKING AND MARKING

### 5.1 Packing

The material shall be packed in suitable containers as agreed to between the purchaser and the supplier.

### 5.2 Marking

The containers shall be securely closed and legibly and indelibly marked with the following information:

a) Name of the material,
b) Net mass of the material,
c) Indication of the source of manufacture,
d) Date of manufacture, and
e) Batch or lot number in code or otherwise.

## 6 SAMPLING

The method of drawing representative samples of the product and the criteria for conformity shall be as prescribed in Annex B.

# ANNEX A

( *Table* 1 )

## METHODS OF TEST FOR ALEURITIC ACID

### A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water ( *see* IS 1070 : 1977 ) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

### A-2 DETERMINATION OF MOISTURE CONTENT

The moisture content shall be determined by the Karl Fischer method as given in IS 2362 : 1973.

### A-3 DETERMINATION OF MOLECULAR MASS

**A-3.1 Procedure**

The molecular mass is determined by the acid value method as given in **15** of IS 6921 : 1973 and calculated as follows.

**A-3.1.1** *Calculation*

$$\text{Molecular mass} = \frac{M}{V \times N}$$

where

$M$ = mass in g of the sample taken,

$V$ = volume in ml of potassium hydroxide ( KOH ), and

$N$ = normality, of potassium hydroxide solution.

### A-4 DETERMINATION OF MELTING POINT

The melting point of the method shall be determined by the method given in IS 5762 : 1970.

### A-5 DETERMINATION OF RELATIVE DENSITY

**A-5.1 Apparatus** — Specific gravity bottle.

**A-5.2 Procedure**

Clean the specific gravity bottle and dry it carefully. Weigh the empty bottle in a balance. Put 2 g of the solid sample in the bottle and weigh again. Now fill the bottle completely with water and replace the stopper carefully so that no air bubble sticks inside Wipe out the bottle and weigh again. Pour out the contents of the bottle, wash it thoroughly and fill it completely with water, wipe the outside of bottle dry and weigh again.

**A-5.3 Calculation**

$$\text{Specific gravity} = \frac{M_2 - M_1}{(M_4 - M_3) + (M_2 - M_1)}$$

where

$M_1$ = 5 mass in 'g' of empty bottle,

$M_2$ = mass in 'g' of bottle with solid sample,

$M_3$ = mass in 'g' of bottle with solid sample and water, and

$M_4$ = mass in 'g' of bottle and water.

### A-6 DETERMINATION OF INFRA-RED SPECTRUM

**A-6.1 Apparatus**

IR spectrophotometer.

**A-6.2 Reagent** — Potassium bromide ( KBr ).

**A-6.3 Procedure**

Intimately grind about 100 mg of pure, dry potassium bromide together with 1.5 mg of solid sample. Fuse the mixture at room temperature under vacuum and compress at high pressure to get a solid pellet. The pellet is mounted on a holder and held in the sample beam path to obtain the spectrum. The absorbance of the sample is compared with that of an approved sample.

**A-6.3.1** The sample shall be considered passing if its spectrum compares closely with that of the approved sample.

# ANNEX B

( *Clause* 6 )

## SAMPLING

### B-1 LOT

All containers of the same size and capacity and containing alcuritic acid produced under similar conditions of manufacture shall constitute a lot.

Each lot snail be tested separately for the various requirements of the specifications.

### B-2 SCALE OF SAMPLING

The number of containers to be selected for the sample shall be as given in Table 2.

**Table 2 Scale of Sampling**

| No. of Containers in the Lot | No. of Containers to be Selected |
|---|---|
| Up to 25 | 3 |
| 26 to 50 | 4 |
| 51 to 100 | 5 |
| 101 to 300 | 7 |
| 301 to 500 | 9 |
| 501 and above | 12 |

These containers shall be selected at random from the lot as per IS 4905 : 1968.

### B-3 PREPARATION OF TEST SAMPLES

From each of these selected containers a representative portion of material shall be taken, which shall be sufficient for carrying out tests for all characteristics given in the specification.

**B-3.1 Composite Sample**

Out of these portions, a small but equal quantity of material shall be taken and mixed thoroughly to make a composite sample.

**B-3.2 Individual Sample**

The remaining portion of the material from each selected container shall constitute an individual test sample.

### B-4 NUMBER OF TESTS

Tests for melting point and specific gravity shall be performed on individual samples. The test for all other characteristics shall be conducted on composite sample.

### B-5 CRITERIA FOR CONFORMITY

The lot shall be deemed to conform to the specifications if all the test results according to **B-4** satisfy the corresponding requirements.

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